Adhesion of resin cement to contemporary hybrid ceramic and polymeric CAD/CAM materials: effect of conditioning methods and ageing

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Adhesion of resin cement to contemporary hybrid ceramic and polymeric CAD/CAM materials:

Effect of conditioning methods and ageing

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Short title: *Resin-cement adhesion to hybrid CAD/CAM materials*

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Keywords: Adhesion; Adhesive cementation; Bond strength test; CAD/CAM; Hybrid materials; Macroshear test; Surface conditioning

Introduction

The advances in the field of ceramics and resin composite materials in restorative dentistry significantly decreased the use of metals in dentistry. This is also in part due to increased implementation of Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) technologies in dentistry. Besides optical expectations, with recent CAD/CAM materials, the lost tooth substances could be restored with materials having similar physical properties compared to that of the dentin and enamel. While ceramics have superior optical properties, they are known to be stiffer and more brittle, which leads to chipping of the veneering ceramics and excessive tooth wear of the antagonistic teeth. On the other hand, although resin composite materials demonstrate low mechanical properties and poor wear resistance, their fabrication is considerably easier and cheaper compared to ceramic materials [1].

The introduction of multiphase materials aim to unify the best mechanical characteristics of resin composites and ceramics. Contemporary CAD/CAM materials are typically based on glassy matrix ceramics (lithium disilicate), polycrystalline ceramics (zirconia), highly filled polymers or sometimes classified as resin nano ceramic (RNC) [2], polymer infiltrated ceramic network (PICN) [3,4] or hybrid ceramics that are usually a mixture of lithium disilicate, alumina and zirconia or aluminium-enriched feldspar-ceramic fused by a polymeric material.

Several studies have investigated the mechanical properties of recent CAD/CAM materials reporting higher modulus of elasticity compared to conventional dental ceramics, lower resistance to wear and thereby being less abrasive to the opposite dentition [5,6]. However, for the longevity of reconstructions and in particular the minimal invasive ones made of hybrid CAD/CAM materials not only the physical properties but also the adhesion of the resin cements to these restorative materials as well as to the

tooth structures is of significant importance [7,8]. Adhesive cementation increases the retention and the fracture resistance of such restorative materials, enhances the marginal adaptation and prevents micro-leakage at the restoration margins. Regarding traditional ceramic and resin composite materials, the literature provides reliable and well established adhesion protocols [9]. Since glassy matrix ceramics (i.e. feldspath, lithium disilicate) are acid-sensitive, they are recommended to be etched with 5 to 9.6% hydrofluoric acid (H) gel. The resulting selective solubility of the silicate phase yields to surface degradation and eventually a topographic relief that favours micro-mechanical retention. Furthermore, the application of a silane coupling agent on the etched ceramic surface increases the chemical bonding between the ceramic and the resin cement [10-12]. This process promotes the cement hydrophilicity on the given ceramic surface [13,14] enhancing the contact with resin cements. In addition, the bifunctional silane coupling agents couple the silica oxides of the ceramics to the organic matrix of the resin cements by means of siloxane bonds [15,16]. As regards to resin composite materials, surface conditioning with air-borne particle abrasion with aluminum-oxide (Al_2O_3) particles has been recommended as the most effective method to roughen the surface reliably [17]. Independent of the indirect resin composite type particle size of about 50 µm, appears to be sufficient to create a sufficiently rough surface [18-25]. In addition, previous studies reported even improved adhesion to resin composites through tribochemical silica coating (CoJet or Rocatec system, 3M ESPE, St. Paul, MN, USA) that provides physicochemical surface conditioning (air-abrasion followed by silanization) often resulting in cohesive failure in the substrate [19,21-23,25,26].

Unlike ceramic and resin composite materials, there is a lack of information regarding the best possible and durable adhesion protocol to be employed in on hybrid materials [27]. Since some hybrid ceramic materials contain no polymer network but a variety of glassy

matrix or polycrystalline ceramic particles, others are based on a mixture of feldspathic ceramic infiltrated with polymer. In fact, both phases require and benefit from different types of surface conditioning. Yet, application of multiple conditioning methods may yield to cross-contamination of the surface by each method. On the other hand, one type of conditioning method be it HF acid etching or air-abrasion could deliver sufficient adhesion. Furthermore, manufacturers` instructions present inconsistent information regarding cementation protocols resulting in confusion among clinicians.

The objectives of this study were therefore to evaluate the effect of two different surface conditioning methods on adhesion of resin cement to different CAD/CAM materials with and without ageing and investigate on the failure types after debonding. The hypotheses tested were that type of surface conditioning method would not show significant effect on adhesion and that ageing would decrease adhesion significantly.

Materials and Methods

The brands, types, manufacturers and chemical compositions of the materials used in this study are listed in Table 1. Distribution of experimental groups based on the CAD/CAM materials, conditioning methods and aging procedure are presented in Fig. 1.

Specimen preparation

Blocks of seven different CAD/CAM materials (N=360, n= 30 per group), namely a) Lithium disilicate (IPS e.max CAD-LIS, Ivoclar Vivadent, Schaan, Liechtenstein), b) Zirconia (IPS e.max ZirCAD-ZIR), Ivoclar Vivadent), c) polymer (Lava Ultimate-LAV, 3M ESPE, Minnesota, USA), d) Polymer infiltrated ceramic (Enamic-ENA, VITA Zahnfabrik, Bad Säckingen, Germany), e) Polymer infiltrated ceramic (Cerasmart270-CS1, GC, Tokyo, Japan), f) Polymer infiltrated ceramic (Cerasmart300-CS2, GC), g) Lithium disilicate reinforced alumina (n!ce-NIC, Institut Straumann AG, Basel, Switzerland) were

cut into slices (3x6x8 mm^3) using a precision cutting machine (Struers Accutom-50, Struers A/S, Ballerup, Denmark).

The specimen surfaces were polished using silicon carbide papers in sequence of 600, 800 and 1200 grit (English Abrasives Ltd, London, United Kingdom) under water cooling and ultrasonically cleaned (Vitasonic, VITA Zahnfabrik) for 5 minutes in distilled water and dried. The specimens were then randomly assigned to the following surface conditioning methods:

Surface conditioning methods

Method 1 (H): Material surfaces were etched with 5% hydrofluoric acid (H) (VITA Ceramic Etch, VITA Zahnfabrik) for 60 s, rinsed with air-water spray and dried. Then one coat of silane coupling agent was applied (Monobond Plus, Ivoclar Vivadent) with a clean microbrush and waited for its reaction for 60 s.

Method 2 (A): Material surfaces were air abraded using chairside silica coating system (CoJet System, 3M ESPE) for 20 s from a distance of approximately 10 mm under a pressure of 2.5 bar.

Group LIS was conditioned with only Method 1and Group ZIR only with Method 2 and acted as the control groups.

Before cementation, a thin layer of adhesive resin (Heliobond, Ivoclar Vivadent) was applied with a clean micro brush, excess adhesive was removed with air and photo-polymerized for 20 s (XL 3000, 3M ESPE; light output: 500 mW/cm^2). Low viscous resin cement (Variolink Esthetic II, Ivoclar Vivadent) was mixed following the manufacturer's instructions and injected into the mould using a syringe (Centrix, DF, Rio de Janeiro, Brazil) and bonded onto the conditioned specimens using a translucent polyethylene mould (inner diameter: 3 mm; height: 5 mm) and photo-polymerized for 40 s from the top and four different sides of each specimen. Then, oxygen inhibiting gel (Oxyguard II,

Kuraray Co. Ltd, Tokyo, Japan) was applied on the free surfaces for 1 min and washed and rinsed.

One half of the specimens was randomly subjected to ageing (thermocycling 5000 cycles, 5-55°, dwell time: 30 s) (Haake DC 10, Sigma-Aldrich, St. Louis in Missouri, USA) and the other half was stored in distilled water at 37°C for 24 h.

Macroshear test

Specimens were mounted in the specimen holder of the Universal Testing Machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany) and the force was applied to the adhesive interface until failure occurred. The load was applied with a 50 kgf load cell to the substrate-adherend interface as close as possible to the surface of the substrate at a crosshead speed of 1 mm/min and the stress-strain curve was analyzed with the software program (TestXpert®, Zwick ROELL, Ulm, Germany).

Failure type evaluation

Failure sites were initially observed using an optical microscope (Zeiss MC 80 DX, Jena, Germany) at x50 magnification. The failure types were classified as follows: Score 1= Adhesive failure at the ceramic-cement interface with no cement remnants left on the substrate; Score 2= <1/3 cement left adhered on the substrate; Score 3= >1/3 cement left adhered on the substrate; Score 4= Cohesive failure within the substrate.

In order to evaluate the qualitative alterations on the substrate surfaces, from each group three additional specimens were prepared (polished-unconditioned, H and A conditioned). After ultrasonic cleaning, they were evaluated under the scanning electron microscopy (SEM). The specimens were coated with gold and palladium sputter and placed in high vacuum. Qualitative examination was performed under a magnification of x5000, at 5 kV (Zeiss Supra V50, Carl Zeiss, Oberkochen, Germany).

Statistical analysis

Statistical analysis was performed using statistical software R [28] including the package ggplot2 (R Foundation for Statistical Computing, Vienna, Austria) [29]. Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. The means of each group were analyz ed using a two-way analysis of variance (2-way ANOVA) and Tukey`s post hoc tests with the shear bond strength (MPa) as the dependent variable and substrate types (7 levels: LIS, ZIR, CS1, CS2, LAV, NIC, ENA), conditioning method (2 levels: H vs A) and aging (2 levels: dry versus thermocycling) as the independent factors. *P* values less than 0.05 were considered to be statistically significant in all tests.

Results

Type of substrate, conditioning method and aging had significant effect on adhesion values ($p<0.05$) (Table 2).

In dry conditions, regardless of conditioning method ENA-H (18.4±3.9), ENA-A (19±4) and CS2-H (15.3±4.) and CS2-A (15.9±3.3) showed no significant difference ($p>0.05$) (Table 3, Fig. 2). NIC-H (18.3±3.1) was also not significantly different compared to these groups ($p>0.05$). In aged conditions, ENA-H, ENA-A, LAV-H, LAV-A, CS2-A (15±4 - 11.6±5) showed no significant difference ($p>0.05$), with the CS2-A (15±4) showing the least reduction (5.5%) compared to all other groups (5.8-62.6%) in adhesion after aging.

While in dry conditions, exclusively cohesive failures were observed in CS1-A, ENA-H and ENA-A, and after aging, CS1-A (100%) followed by ENA-H, ENA-A (93%), CS2-A (80%) presented the highest incidence of cohesive failures (Table 2).

SEM microphotographs of the substrate surfaces (x5000) at baseline (control-C) indicated the rougher surfaces with porosities with ENA followed by CS2, LAV and CS1 compared to other materials. After hydrofluoric acid etching (H) ,ENA material showed again distinct etched pattern followed by LAV and LIS demonstrating dissolved areas of the matrix. After

silica-coating (A) however, the substrate surfaces showed evident roughened surfaces with the impact of sand particles offsetting the variations in microtexture observed at baseline (Figs. 3a-u).

Discussion

CAD/CAM materials of hybrid nature, be it polymeric or ceramic, differ greatly in their composition where surface conditioning method dictates the adhesion of resin cements to such materials. This study undertaken in order to evaluate the effect of two different commonly applied surface conditioning methods, namely hydrofluoric acid etching (H) and silica coating followed by silanization (A) on the adhesion of resin cement to various CAD/CAM materials with and without aging. Based on the results of this study, since surface conditioning method type significantly affected the results, the first hypothesis could be rejected. On the other hand, aging decreased the bond strength results significantly, yielding to acceptance of the second hypothesis.

In order to measure the bond strength values between an adherent and a substrates, a number of test methods could be employed. Overall, adhesion related studies in dentistry, require bonded surface areas ranging from 3 mm^2 to 1 mm^2 in macro- and micro-test methods, respectively [30,31]. Due to the reduced bonded area and more homogeneous distribution of stresses, micro-test methods tend to show significantly higher bond strength results than the macro-test methods which could eventually affect the ranking of materials being tested in one study [31,32]. In this study, macroshear test method was chosen considering the polymeric and ceramic phases present in one materials such as in the case of ENA. The larger bonded surface area would involve the adhesion of the resin both to the polymeric and the ceramic component of the material. Similarly, in the case of LAV for instance the polymeric network contains high amount of inorganic fillers and adhesion of the

resin cement to both phases were expected to differ on one substrate. Information derived from macroshear tests was expected to tackle the problem of adhesion aspects to multiphase materials as by definition they cover a larger bonded surface area.

In this study, for highly filled polymer (LAV), polymer infiltrated ceramic network (ENA), polymer infiltrated ceramic (CS1 and CS2) and lithium disilicate reinforced alumina (NIC), surface conditioning with chairside silica coating followed by silanization, resulted similar or even better bond results compared to H conditioning method. In fact, H is a well-established method, for dissolving the silica phase of the glassy matrix ceramics but polycrystalline ceramics show better bond strength after surface roughening by air particle abrasion and silanization [9]. Yet, among the materials LAV, ENA, CS1 and CS2 presenting both organic and inorganic phases did not benefit from H application only. Although SEM images showed distinct solubility of the inorganic phase after H application, surface conditioning with A provided similar or better adhesion results, especially after aging conditions. It could be anticipated that the coating encompassing alumina and silica particles allowed for more durable siloxane bonds on the coating when compared to their adhesion to inorganic phase after H only. Furthermore, the voids created after H application were most likely more prone to water penetration and thereby, more hydrolytic degradation after thermocycling [33]. The percentage decrease of adhesion with CS1-H (35.5) CS2-H (47.4), LAV-H (10.6), ENA-H (31.7) clearly supports this assumption. In a previous study [34] where microshear test was employed, H method applied on LAV, ENA, CS did not improve adhesion of resin cement compared to non-conditioned groups after 5000 cycles of thermocycling. In that study, ENA (8.7) showed significantly higher bond strength values compared to CS (7.6) and LAV (7.2). Yet, the difference was only within the range of 1 to 1.5 MPa. Although no conditioning was not practiced in our study, the findings of this previous study, partially confirms less effect obtained from H on multiphase materials since in some groups H and A presented no

significant difference (LAV and ENA). In another study where microtensile test was used, ENA after H method delivered high bond strength values (23.4) being not significantly different to that of A (15.7) [35]. Similarly, LAV presented significantly higher bond strength with A (16.8) compared to H only (11.2) after 10.000 cycles of thermocycling. It has to be noted that the standard deviations in that study were higher than those observed in our study which could be attributed to above mentioned issues related to the test methods. Moreover, the pretest failures during cutting were not involved in that study [35]. In this study, no pretest failures were experienced during thermoycyling. Nevertheless, considering the hazardous compounds, present in H and concerns on clinical applications of such agents chairside [36], the A conditioning method appears to offset the variations in substrate characteristics [3,33,36-38]. In addition, moderate roughening with 30 µm silica particles apparently delivers better adhesion as supposed to 110 µm Al_2O_3 for conditioning organic and inorganic materials [39,40] where also the latter yields to more frequent adhesive failures [41]. The manufacturer's instructions of ENA suggests the use of 5% H etching prior to cementation whereas the instructions for LAV suggests the A method. However, based on the high degradation rate after H application on especially for CS1 and CS2, clinicians may consider using the A method only for these materials.

Bond strength results in adhesion studies should be also interpreted with failure types. Cohesive failures in the substrate indicate that bond strength of the adhesive system and the resin cement exceeds that of the cohesive strength of the substrate. In this study, after aging, CS1-A (100%) followed by ENA-H, ENA-A (93%), CS2-A (80%) presented the highest incidence of cohesive failures, presenting more reliable adhesion results coupled with the bond strength results.

Among tested CAD/CAM materials, NIC was a lithium disilicate reinforced alumina where neither H nor A resulted in high bond strength values. Moreover, the hydrolytic degradation

with this material was the highest especially after H compared to all tested materials. The manufacturer of this material suggest the use of H but clinicians should note the decrease in adhesion after this conditioning method in conjunction with this material.

One interesting finding of this study was the non-significant adhesion values between the non-aged and aged ZIR groups. Typically, hydrolytic degradation of methacrylate or phosphate monomer base adhesive resin has been evident in numerous previous studies [42]. One reason for the stable bond to ZIR in this study could be attributed to higher degree of polymerization of the tested resin cement where the photo-initiator has been changed from conventional camphorquinone to lucirin which certainly needs further investigation.

Reflecting the increase in digital applications and intention to produce tooth-like materials with organic and inorganic phases in dentistry, clinicians should consider the corresponding conditioning method prior to adhesive cementation. In this regard, manufacturer's instructions are not well- established for all materials and attempts are being made to avoid the use of H clinically or at the lab site [36]. In this context, silica coating and silanization could be a more general option to condition CAD/CAM materials of organic or inorganic origin or a combination of them both, providing that hydrolytic degradation is still unavoidable with either H and A at varying rates which needs to be clinically confirmed.

Conclusions

From this study, the following could be concluded:

1. The adhesion of the tested resin cement to the CAD/CAM materials was influenced by the surface conditioning method type, ageing and the type of substrate.

2. For highly filled polymer (LAV), polymer infiltrated ceramic network (ENA), polymer infiltrated ceramic (CS1 and CS2) and lithium disilicate reinforced alumina (NIC), surface

conditioning with chairside silica coating followed by silanization, resulted similar or better bond results compared to hydrofluoric etching and silanization.

3. After aging, reduction in bond strength was observed in all cement-material combinations with the least for CS2 after silica coating and silanization. Aging resulted in more hydrolytic degradation in hydrofluoric acid etched groups as opposed to silica coating and silanization.

4. After silica coating and silanization similar or higher incidence of cohesive failures were evident compared to hydrofluoric etching and silanization after aging, except for NIC.

Clinical Relevance

Considering higher bond strength values and high incidence of cohesive failure types, overall, either polymeric or ceramic contemporary multiphase hybrid CAD/CAM materials could be conditioned with silica coating and silanization prior to adhesive cementation which could substitute the use of hydrofluoric etching and silanization.

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Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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Captions to tables and figures:

Tables:

Table 1. The brands, chemical compositions, manufacturers, and batch numbers of the main materials used in this study. Al_2O_3: Aluminium oxide, B_2O_3: Boron trioxide, bis-GMA: bisphenol-A diglycidylmethacrylate, bis-EMA: 2,2-bis(4-(2-methacryl-oxyethoxy)phenyl)propane, bis-MEPP: 2,2-bis(4-methacryloxypolyethoxyphenyl)propane, CO: camphorquinone, DMA: Apliphatic dimethacrylate, DMAEMA: 2-dimethylaminoethyl methacrylate, EBPDMA: ethoxylated bis-GMA, F_3Yb: Ytterbium trifluoride, GPDM: Glycerol phosphate dimethacrylate, HEMA: 2-hydroxyethyl methacrylate, HF: Hydrofluoric acid, HfO_2: Hafnium oxide, K_2O: Potassium oxide, Li_2SiO_3: Lithium metasilicate, Na_2O: Sodium oxide, SiO_2: Siliciumdioxid, TEGDMA: Triethylene glycol dimethacrylate, UDMA: Urethane dimethacrylate, Y_2O_3: Yttriumoxide, ZrO_2: Zirconium dioxide.

Table 2. *P* values indicating significant differences between the mean shear bond strength of the resin cement to the substrate types (MPa) in dry and aged conditions. For groups abbreviations see Fig. 1.

Table 3. The mean shear bond strength and standard deviations of the resin cement to the CAD/CAM materials (MPa ± standard deviations) after two surface conditioning methods (H: Hydrofluoric acid and silanization; A: Chairside silica-coating and silanization), minimum and maximal values (CI 95%) and distribution and frequency of failure types per experimental group analyzed after bond strength test: Score 1= Adhesive failure at the ceramic-cement interface with no cement remnants left on the substrate; Score 2= <1/3 cement left adhered on the substrate; Score 3= >1/3 cement left adhered on the substrate; Score 4= Cohesive failure within the substrate. For significant differences between the experimental groups see Table 2 ($p<0.05$) and for groups abbreviations see Fig. 1

Figures:

Fig. 1. Experimental sequence and allocation of groups depending on the material type and surface conditioning.

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Figs. 3a-u. SEM microphotographs of the substrate surfaces (x5000) at baseline (control-C) **a-g)** Note the ENA material showing rougher surface with porosities followed by CS2, LAV and CS1 compared to other materials, after hydrofluoric acid etching (H) **h-m)** Note again that ENA material followed by LAV and LIS presenting etched patterns with dissolved areas of the matrix, after silica-coating (A) **o-u)** Note the coverage of the substrate surfaces with sand particles offsetting the variations in microtexture observed at baseline.

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Brand	Chemical Composition	Manufacturer	Batch#
IPS e.max CAD	Matrix: glassy phase, 40% Li_2SiO_3 crystals (0.2 to 1.0 µm)	Ivoclar Vivadent, Schaan, Liechtenstein	W05153
IPS e.max ZirCAD	Pre-sintered yttrium-stabilized zirconium oxide blocks, ZrO_2, HfO_2, Al_2O_3, Y_2O_3 and other oxides	Ivoclar Vivadent	I24000O
Lava Ultimate	Matrix: bis-GMA, UDMA, bis-EMA, TEGDMA Filler: 80 wt%, silica (20 nm), zirconia (4-11 nm)	3M ESPE, Minnesota, USA	N938003
VITA Enamic	Matrix: 14 wt%, UDMA; TEGDMA Filler: 86 wt%, SiO_2 (58-63%), Al_2O_3 (20-23%), Na_2O (9-11%), K_2O (4-6%), B_2O_3 & ZrO_2 (<2%)	VITA Zahnfabrik, Bad Säckingen, Germany	65440
Cerasmart270	Matrix: UDMA, bis-MEPP Filler: SiO_2, Barium glass	GC, Tokyo, Japan	1801161
Cerasmart300	Matrix: UDMA, bis-MEPP Filler: SiO_2, Barium glass	GC	1709192
n!ce	Lithium disilicate reinforced lithium aluminosilicate glass ceramic	Institute Straumann AG, Basel, Switzerland	MW273
Vita Ceramic Etch	1ml 0.047 g hydrofluoric acid	VITA Zahnfabrik, Bad Säckingen, Germany	V12791
CoJet	Silica coated alumina, particle size: 30 µm	3M ESPE	664075
Monobond Plus	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	Ivoclar Vivadent	W32661
Heliobond	bis-GMA (60 wt%) TEGDMA (40 wt%)	Ivoclar Vivadent	V14836
Variolink esthetic	Monomer matrix: UDMA, methacrylate monomers Inorganic filler: F_3Yb, spheroid mixed oxide, 38 wt%; particle size: 0.04-0.2 µm (mean: 0.1 µm)	Ivoclar Vivadent	W95565 W07567
Oxyguard II	Glycerol 50-70 wt%, polyethylene-glycol, catalysts, accelerators, dyes	Oxyguard II, Kuraray Co. Ltd, Tokyo, Japan	1T0040

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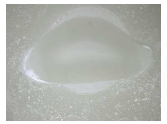
Aged / Dry	LIS-H	ZIR-A	LAV-H	LAV-A	ENA-H	ENA-A	CS1-H	CS1-A	CS2-H	CS2-A	NIC-H	NIC-A
LIS-H		0.007348	n.s.	n.s.	n.s.	n.s.	0.007233	n.s.	n.s.	0.026670	n.s.	0.000643
ZIR-A	n.s.		n.s.	n.s.	n.s.	n.s.	0.000000	0.011732	0.000000	n.s.	0.000000	0.000000
LAV-H	n.s.	n.s.		n.s.	n.s.	n.s.	0.000450	n.s.	n.s.	n.s.	0.005578	0.000028
LAV-A	n.s.	n.s.	n.s.		n.s.	n.s.	0.000007	n.s.	0.007176	n.s.	0.000142	0.000000
ENA-H	0.000429	n.s.	0.005293	0.020081		n.s.	0.000015	n.s.	0.011865	n.s.	0.000265	0.000001
ENA-A	0.000064	0.037751	0.000981	0.004359	n.s.		0.000003	n.s.	0.003832	n.s.	0.000066	0.000000
CS1-H	n.s.	0.013728	n.s.	n.s.	0.000000	0.000000		0.004449	n.s.	0.030000	n.s.	n.s.
CS1-A	n.s.	n.s.	n.s.	n.s.	0.021139	0.004621	n.s.		n.s.	0.040323	0.038810	0.000368
CS2-H	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	0.001343	n.s.		0.000002	n.s.	n.s.
CS2-A	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	0.000184	n.s.	n.s.		0.000000	0.000000
NIC-H	0.000582	n.s.	0.006892	0.025408	n.s.	n.s.	0.000000	0.026713	n.s.	n.s.		n.s.
NIC-A	n.s.	0.000477	0.021756	0.005790	0.000000	0.000000	n.s.	0.005467	0.000030	0.000003	0.000000	

Table 2. *P* values indicating significant differences between the mean shear bond strength of the resin cement to the substrate types (MPa) in dry and aged conditions. For groups abbreviations see Fig. 1.

Groups	Number of specimen	Bond strength (Mean ± SD) (n=15 per group)		Reduction in bond strength after aging (%)	*Min-Max (95% CI)*		Failure Type Distribution n (%)							
							Score 1 		Score 2 		Score 3 		Score 4 	
		Dry	Aged		Dry	Aged	Dry	Aged	Dry	Aged	Dry	Aged	Dry	Aged
LIS-H	30	12.2±5	10.8±5.1	-11.36	5.89-20.24 (9.38-14.9)	5.94-26.84 (7.95-13.6)	13 (87)	14 (93)	6 (50)	1 (7)	2 (13)	0 (0)	0 (0)	0 (0)
ZIR-A	30	14.4±3.8	15.5±4.7	+7.57	8.07-21.54 (12.3-16.5)	7.04-24.05 (12.9-18.1)	15 (100)	15 (100)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
LAV-H	30	13±2.4	11.6±4.5	-10.6	8.96-17.42 (11.7-14.3)	7.27-25.05 (9.12-14.2)	4 (27)	10 (67)	0 (0)	1 (7)	0 (0)	0 (0)	11 (73)	4 (27)
LAV-A	30	13.5±3.2	12.7±1.8	-5.84	8.72-19.53 (11.8-15.3)	8.66-16.69 (11.7-13.7)	1 (7)	2 (13)	1 (7)	3 (20)	0 (0)	0 (0)	13 (87)	10 (67)
ENA-H	30	18.4±3.9	12.6±3.3	-31.74	10.92-24.89 (16.2-20.6)	5.1-17.61 (10.8-14.4)	0 (0)	1 (7)	0 (0)	0 (0)	0 (0)	0 (0)	15 (100)	14 (93)
ENA-A	30	19±4	13±2.1	-31.8	13.95-29.78 (16.8-21.2)	8.4-16.9 (11.8-14.1)	0 (0)	0 (0)	0 (0)	1 (7)	0 (0)	0 (0)	15 (100)	14 (93)
CS1-H	30	9.4±5.4	6.1±2.2	-35.5	3.24-19.24 (6.38-12-4)	2.06-9.72 (4.86-7.24)	9 (60)	7 (47)	5 (33)	5 (33)	1 (7)	3 (20)	0 (0)	0 (0)
CS1-A	30	13.6±2.3	10.9±2.8	-19.33	9.17-17.75 (12.3-14.8)	6.85-15.64 (9.39-12.5)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	15 (100)	15 (100)
CS2-H	30	15.3±4.2	8±2.2	-47.4	7.51-21.53 (12.9-17.6)	4.6-11.83 (6.79-9.25)	11 (73)	5 (33)	3 (20)	9 (60)	1 (7)	1 (7)	0 (0)	0 (0)
CS2-A	30	15.9±3.3	15±3.7	-5.46	9.94-23.23 (14-17.7)	10.62-21.84 (13-17.1)	1 (7)	0 (0)	0 (0)	3 (20)	1 (7)	0 (0)	13 (87)	12 (80)
NIC-H	30	18.3±3.1	6.8±2.9	-62.63	14.02-23.68 (16.6-20)	3.67-13.3 (5.22-8.44)	3 (20)	7 (47)	1 (7)	3 (20)	0 (0)	2 (13)	11 (73)	3 (20)
NIC-A	30	8.2±2.3	5.3±2	-35.33	3.2-11.41 (6.92-9.45)	2.38-9.64 (4.16-6.42)	4 (27)	10 (67)	5 (33)	5 (33)	1 (7)	0 (0)	5 (33)	0 (0)

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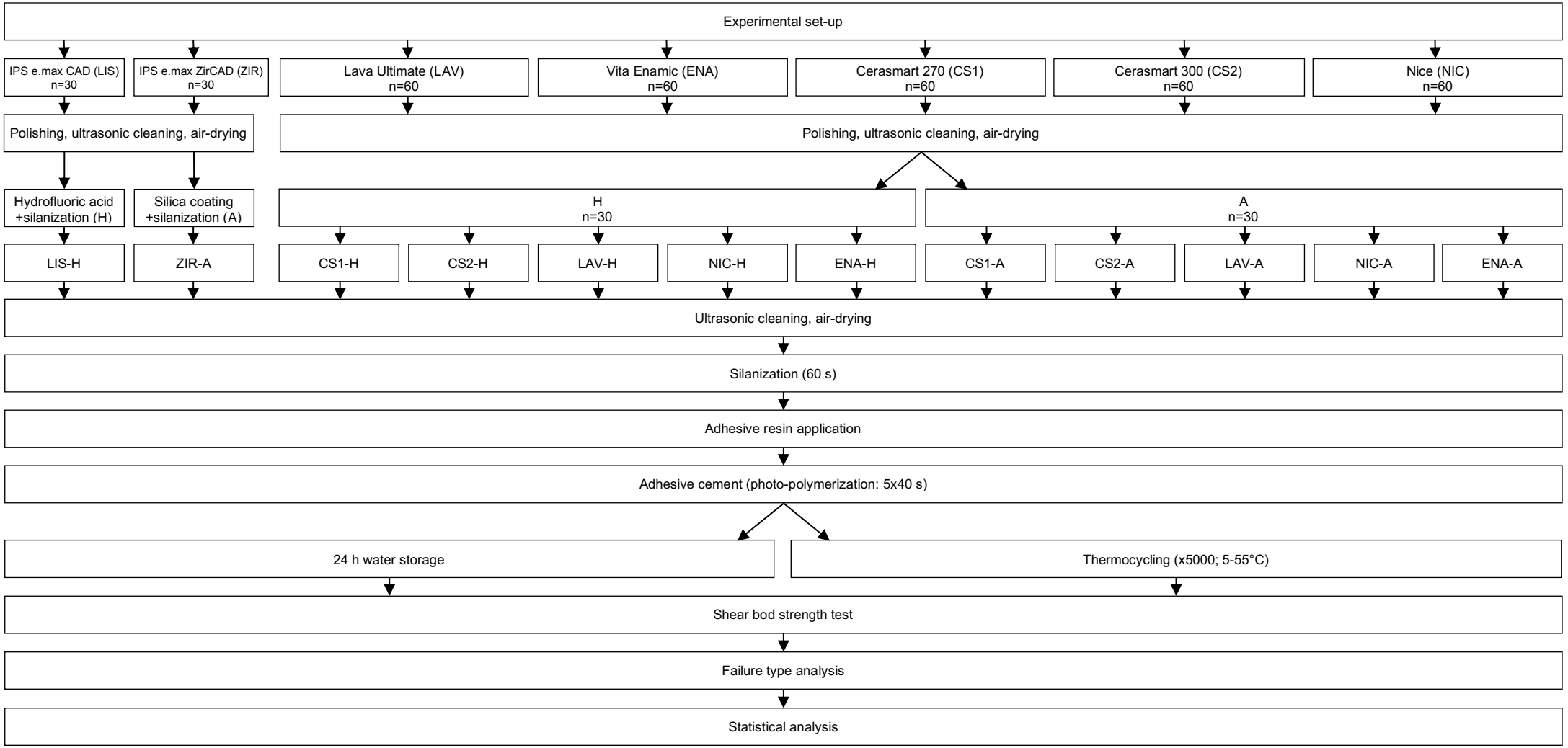
Figures:



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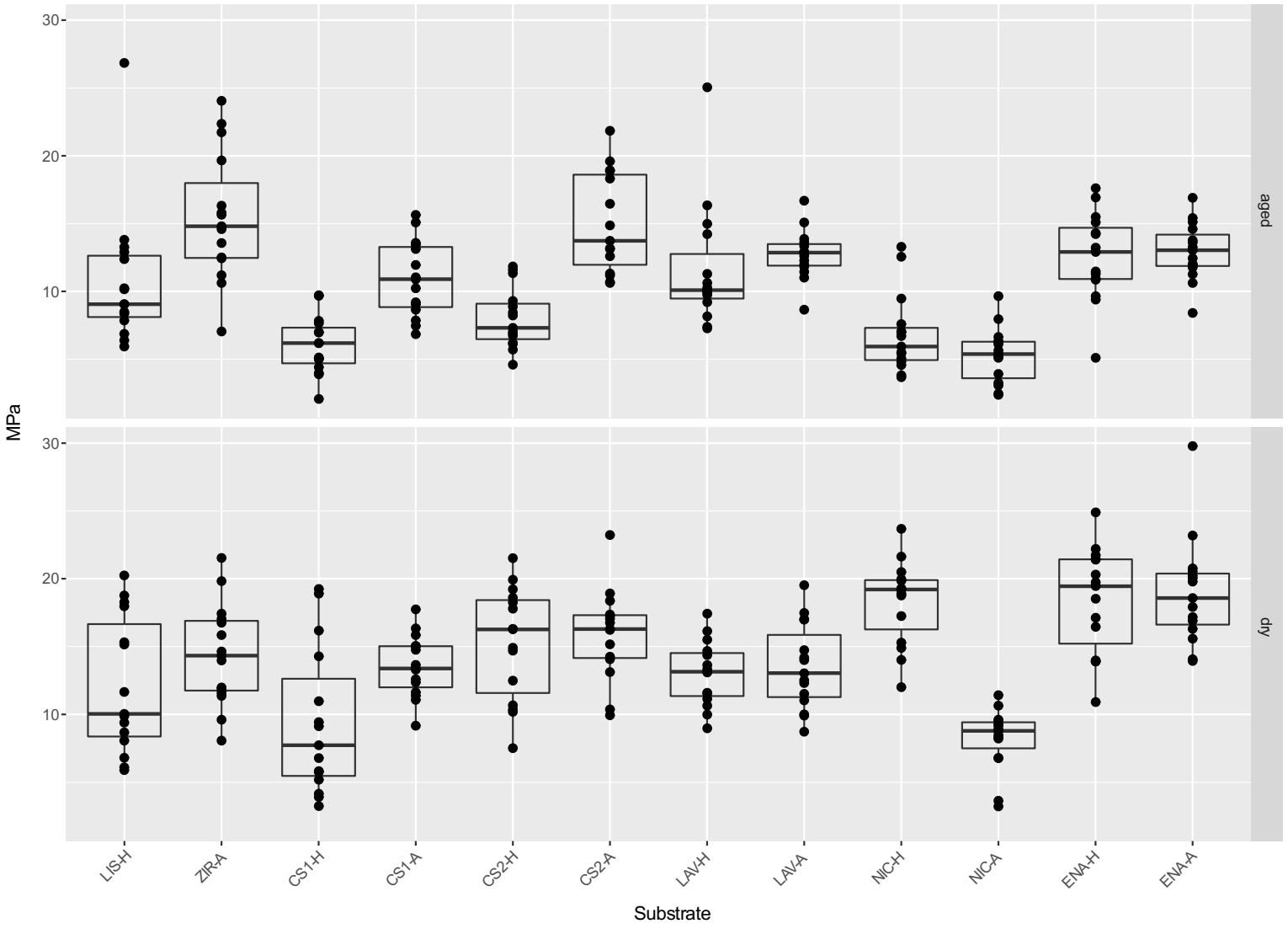

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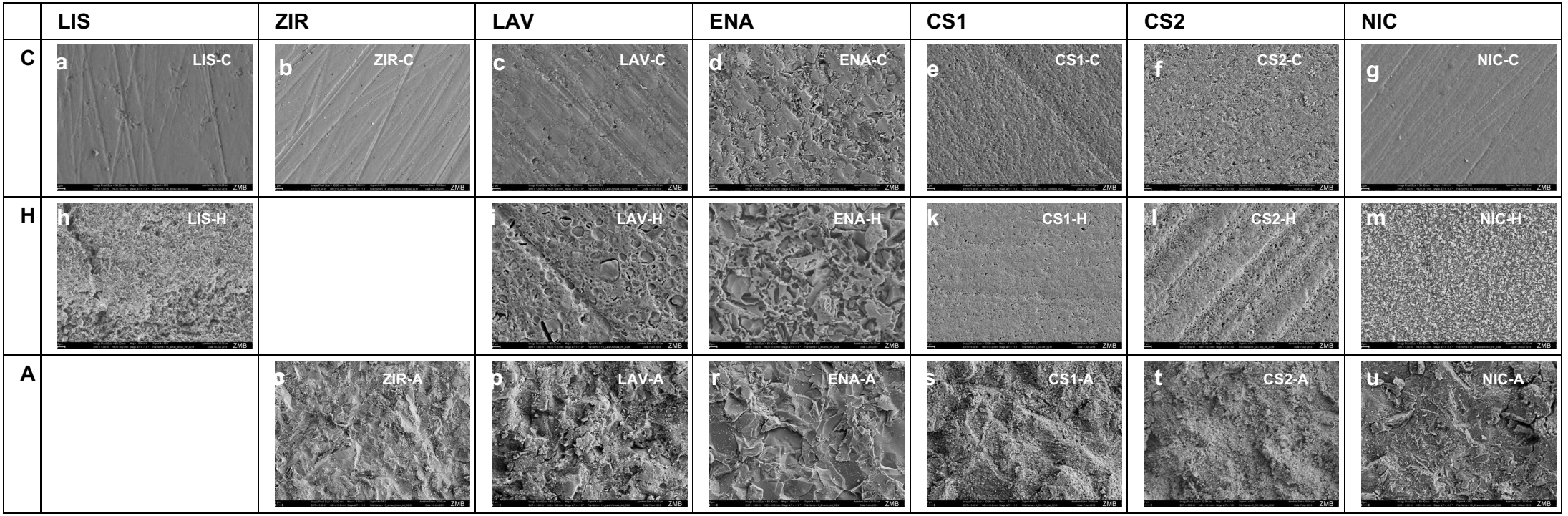

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